

FIG. 1

MORALES, KLR, ETH-SIOH-1 IN CDC13

EXPL S3PUL

SAMPLE		DEC. & VT	
DATE	FEB 27 98	DFRQ	499.699
SOLVENT	CDC 13	DN	HL
FILE	EXP	DPWR	20
ACQUISITION		DOF	6
STFRQ	499.699	DM	NNN
TN	111	DMM	C
AT	3.277	DMF	200
NP	39296	DSEQ	
SW	5996.1	DRES	1.0
FB	3400	HOMO	N
BS	16	DEC2	
TPWR	63	DFRQ2	0
PW	4.7	DN2	
DL	0	DPWR2	1
TOF	0	DOF2	0
NL	400	DM2	N
CT	160	DMM2	C
ALOCK	N	DMF2	200
GAIN	NOT USED	DSEQ2	
FLAGS		DRES2	1.0
11	N	HOMO2	N
LN	N	PROCESSING	
DP	Y	16	6.30
HS	NN	WTFILE	
DISPLAY		PROC	FT
SP	-138.2	FN	NOT USED
WP	5133.1	MATH	R
V\$	8848		
SC	0	WERR	
WC	250	WEXP	
NIMM	20.53	WBS	
LS	33.57	WNT	
RFL	4131.0		
RFP	3627.8		
TH	7		
INS	1.000		
NM	PH		

FIG. 1A

09971857-100301

FOE001" 25812660

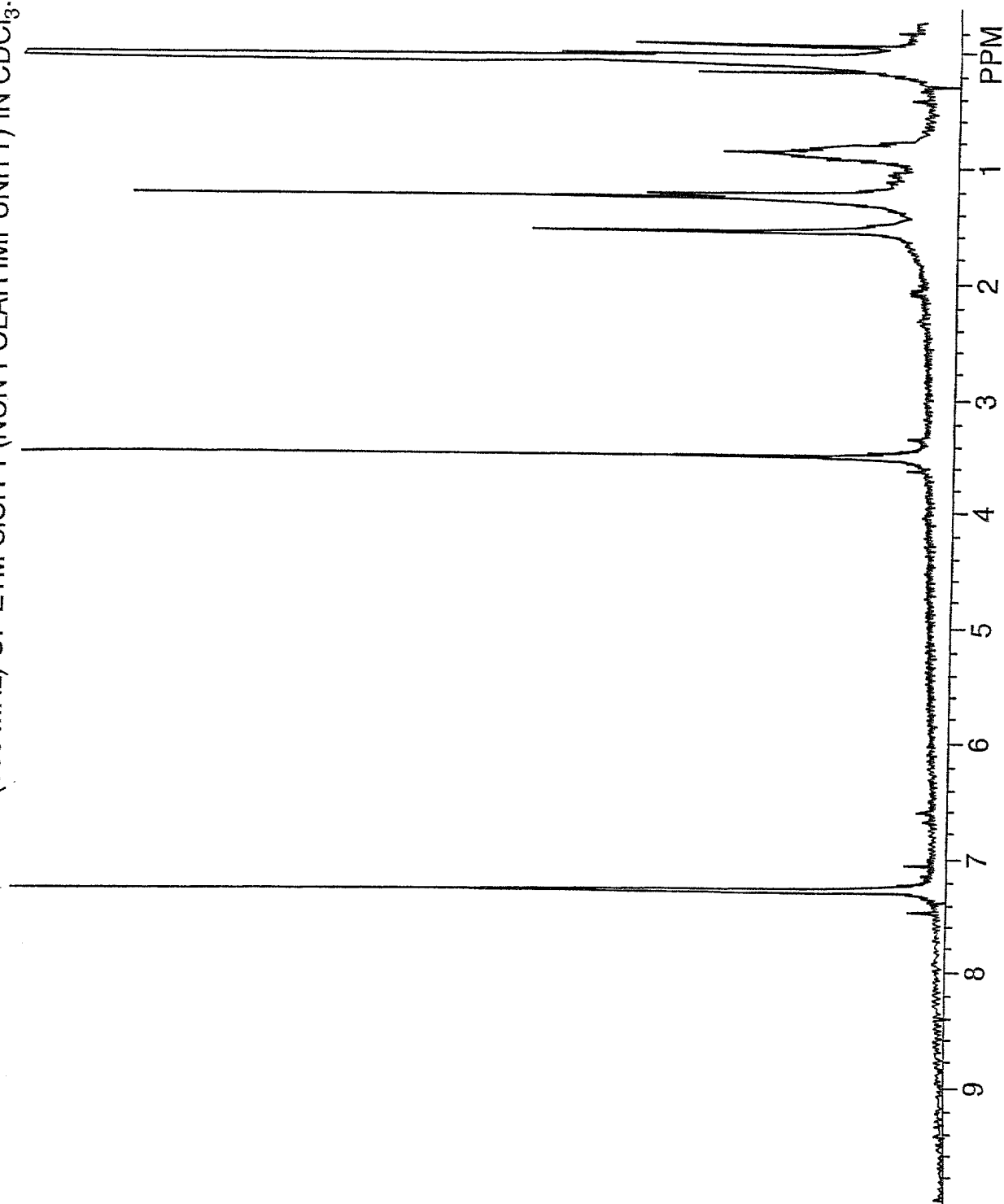
H NMR SPECTRUM (500 MHz) OF ETM-SIOH-1 (NON-POLAR IMPURITY) IN  $\text{CDCl}_3$ .

FIG. 1B

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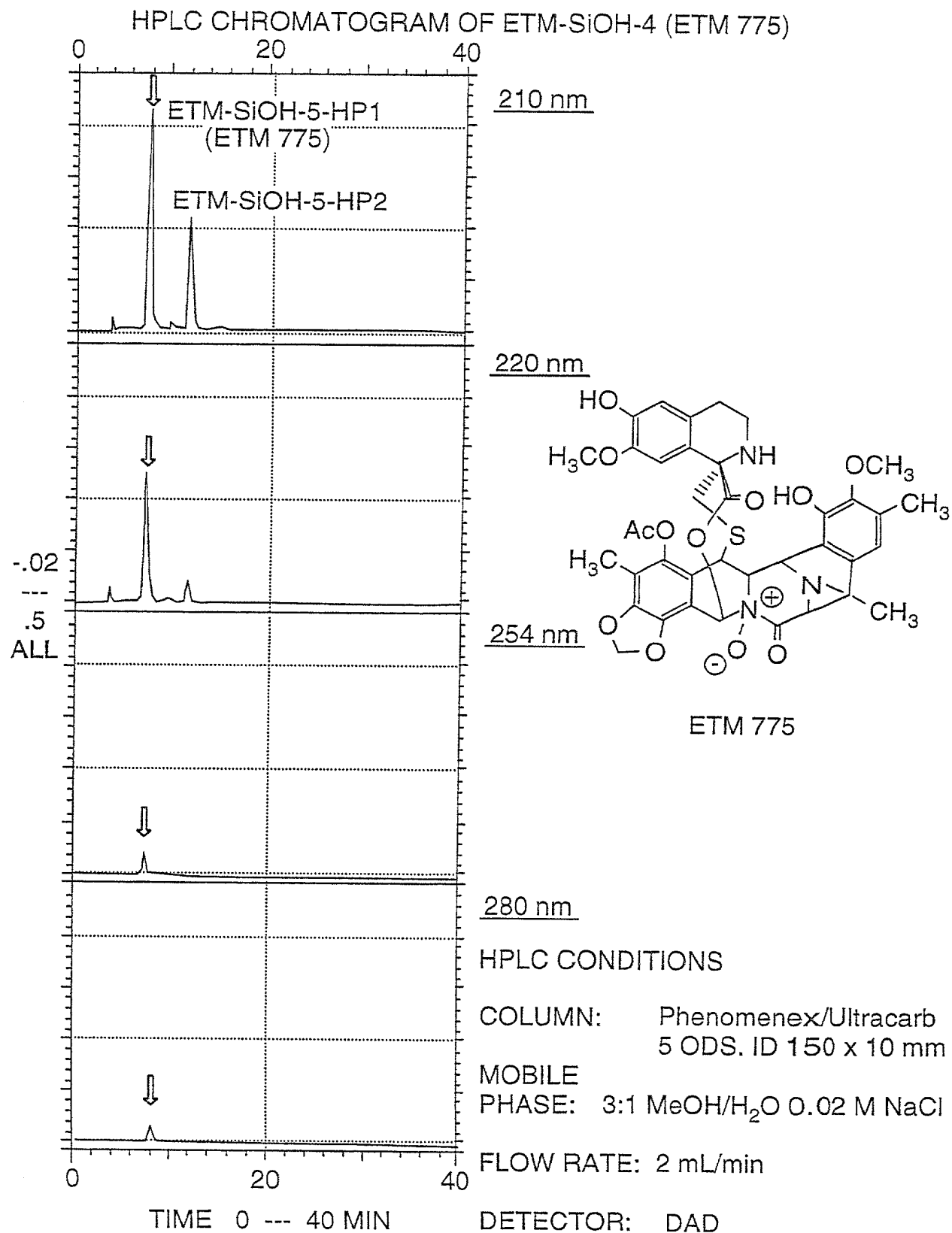


FIG. 2

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# HPLC CHROMATOGRAM OF ETM-SiOH-3 (ETM 305)

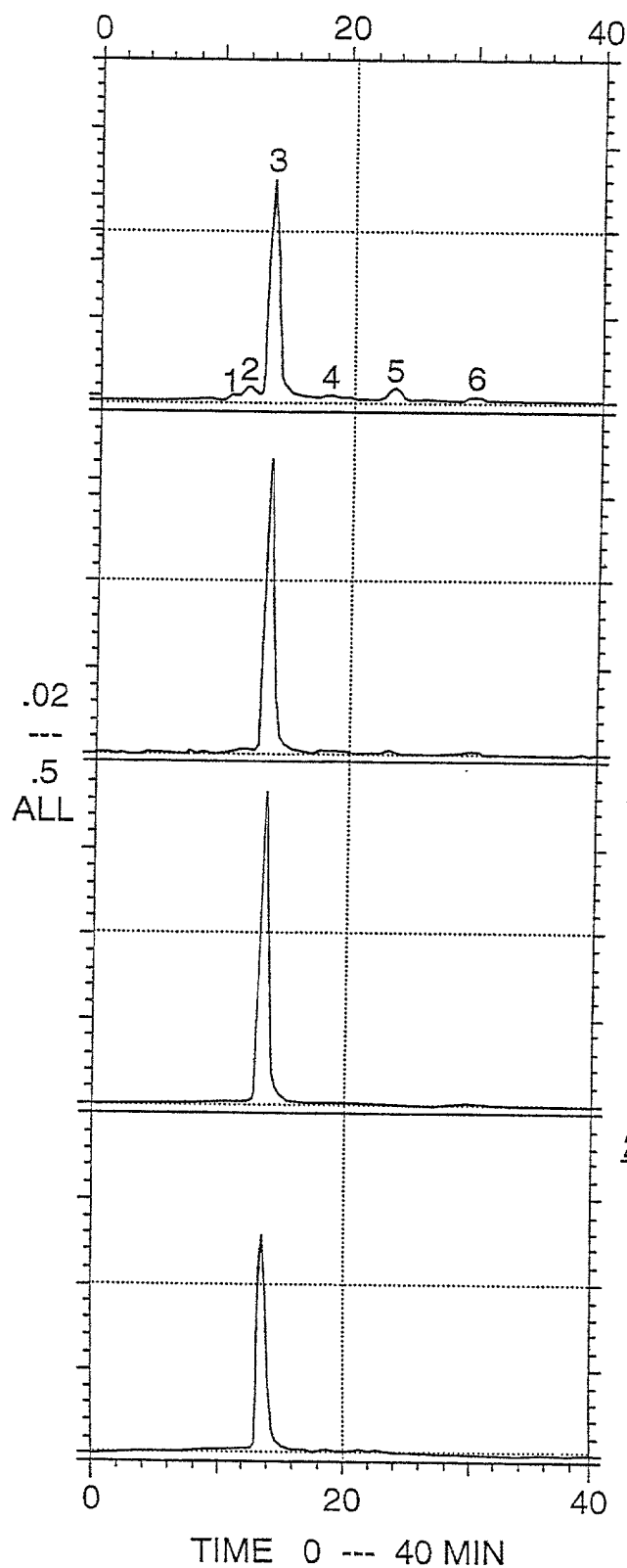
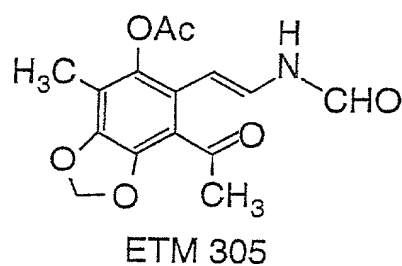


FIG. 3



## HPLC CONDITIONS

COLUMN: Phenomenex/Ultracarb  
5 ODS. ID 150 x 10 mm

MOBILE

PHASE: 3:1 MeOH/H<sub>2</sub>O 0.02 M NaCl

FLOW RATE: 1mL/min

DETECTOR: DAD

-.02  
---  
.15  
ALL



MOBILE

PHASE: 3:1 MeOH/H<sub>2</sub>O 0.02 M NaCl

FLOW RATE: 1mL/min

DETECTOR: DAD

FIG. 4

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LRFAB MASS SPECTRUM OF ETM 305 IN M.B. (MAGIC BULLET<sup>4</sup>)

FILE: ETMSIOH4HP1 IDENT:2 ACQ: 16-DEC-1997 12:43:40 +0:45 CAL:CSI121697

ZAB-SE FAB+ MAGNET BpV: 15.3V TIC:2105487744 FLAGS:HAL

FILE TEXT: MORALES ETM-SIOH-4-HP1 IN M.B.

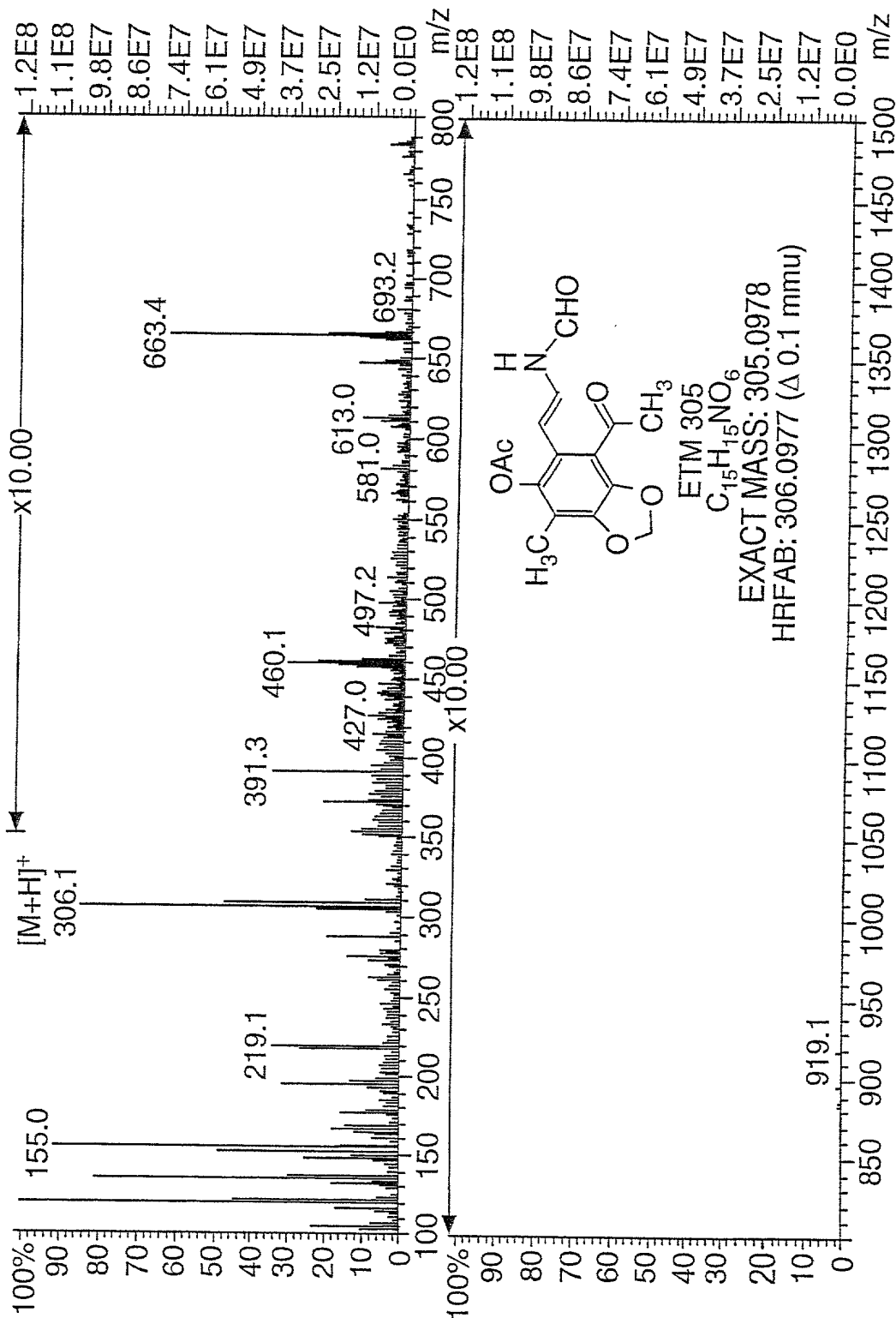


FIG. 5

ESI MASS SPECTRUM OF ETM 305.

etm-sioh-4hp1  
Q5689 7 (1.192) Sm(Mn, 2x0.40): Cm (5:9-1:4)

Scan ES+  
1.91e6

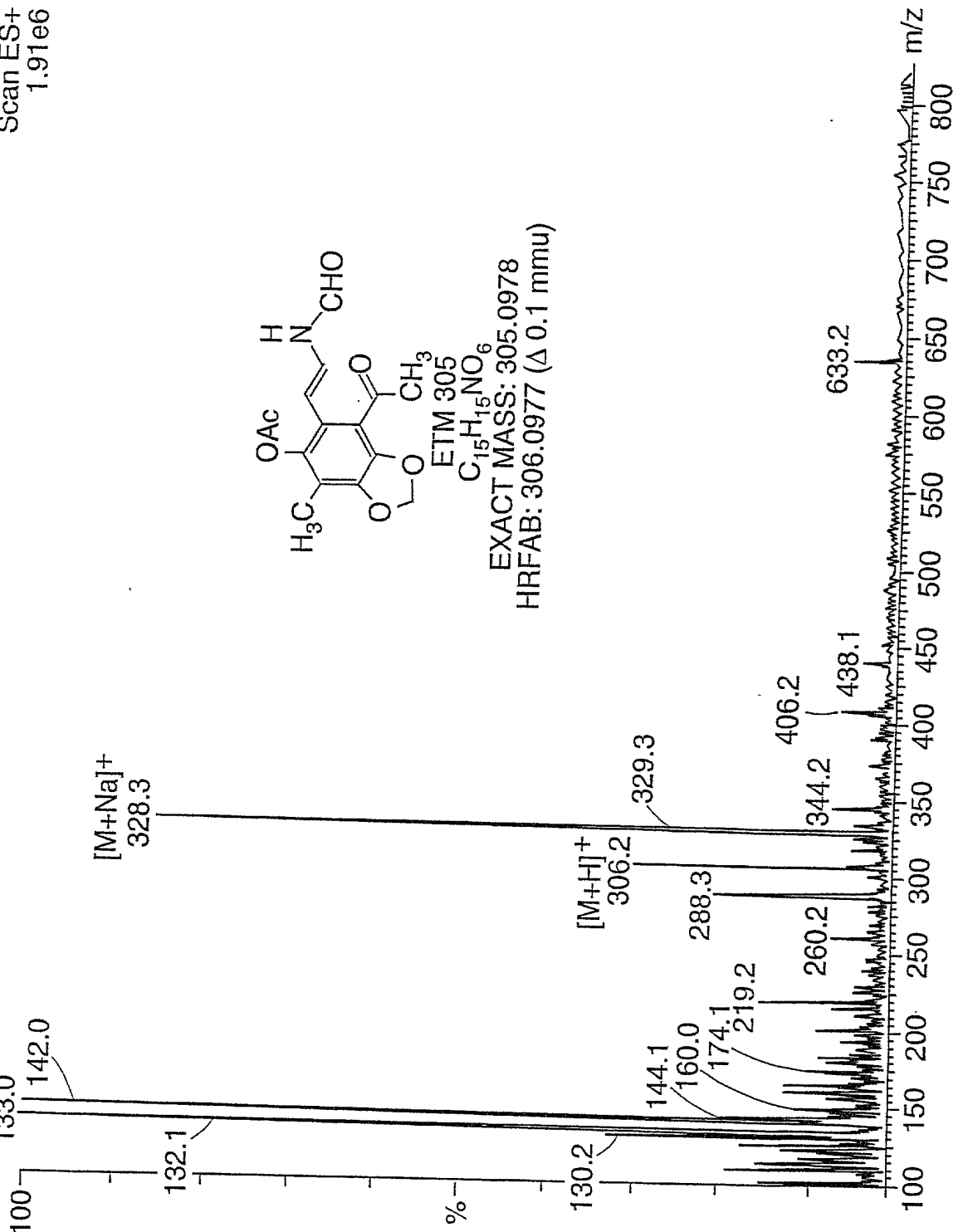
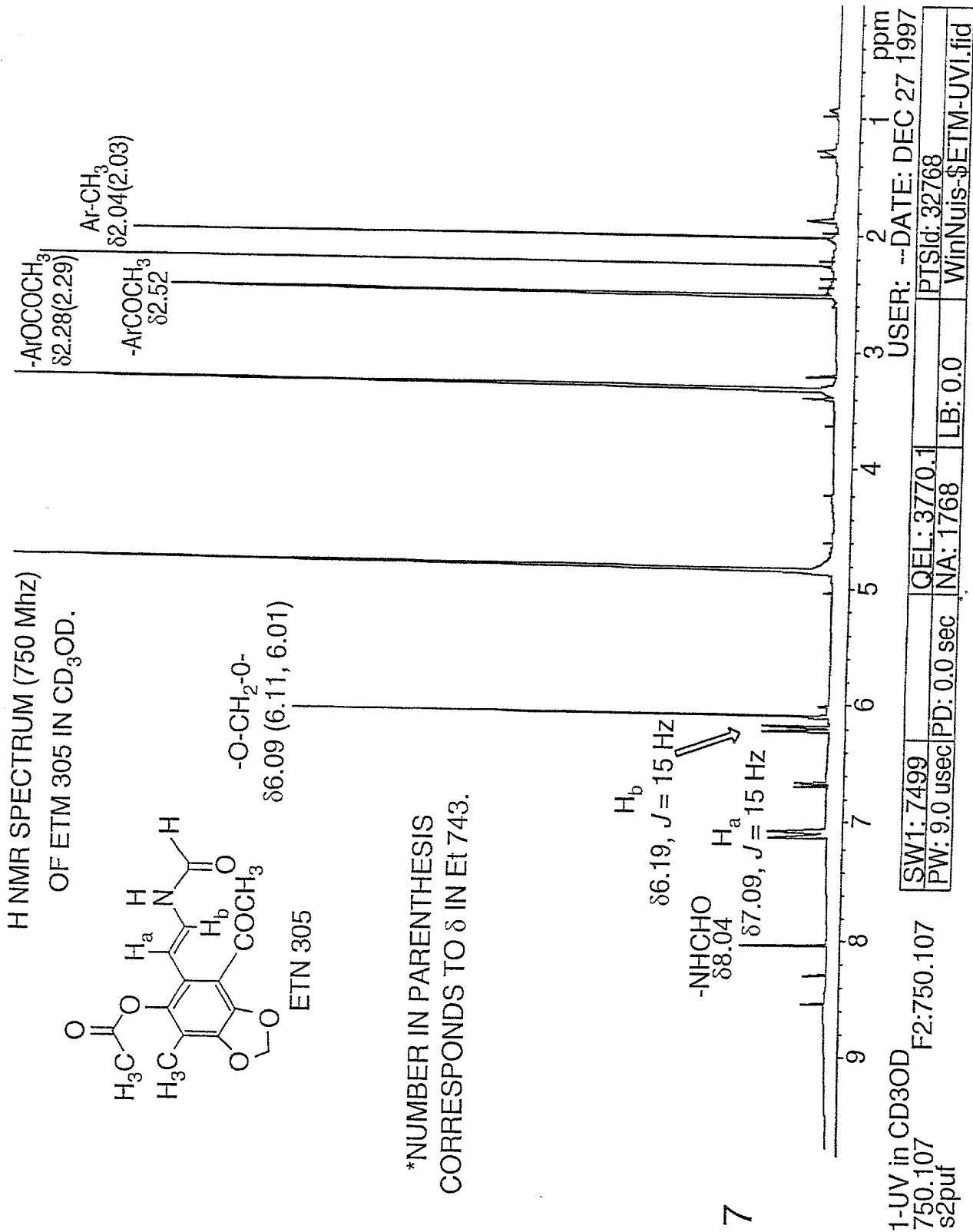
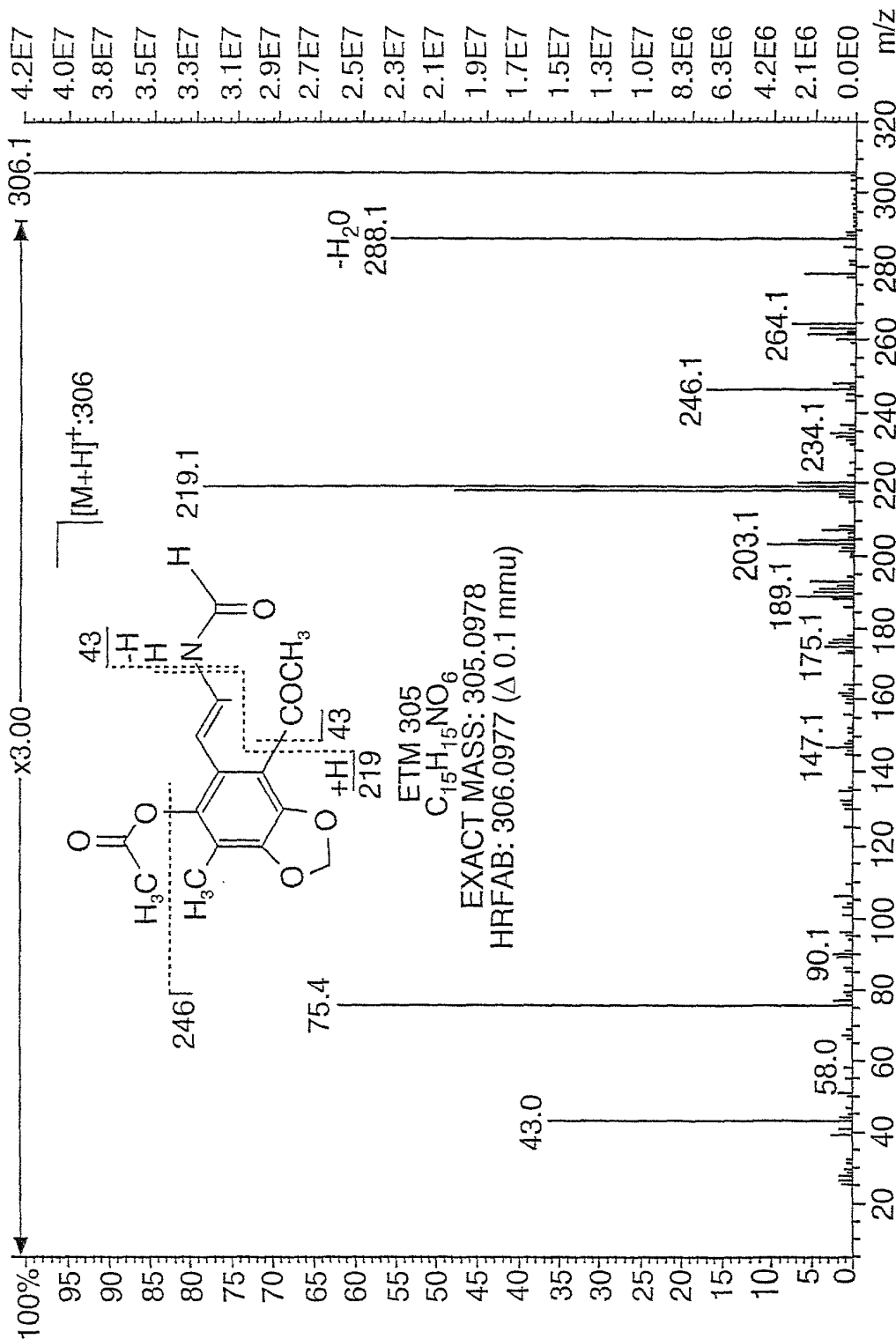


FIG. 6





FILE:MS9289 IDENT:1 SMO(1,7) PKD(7,3,7,0.00%,0.0,0.00%,F,F) SPEC(HEIGHTS, CENTROID) ACQ:13-FEB-1>  
70-4SE FAB+ E2B2 BpM:306 BpV:31.1V TIC:124301384 FM:306.10 FLAGS:NORM  
FILE TEXT: MORALES ETM-UV MSMS ON 306.1 CELL 0.5 HE 90% MORALES ETM-UV MSMS ON 306.1 CELL 0.5 HE>



8  
G.  
F

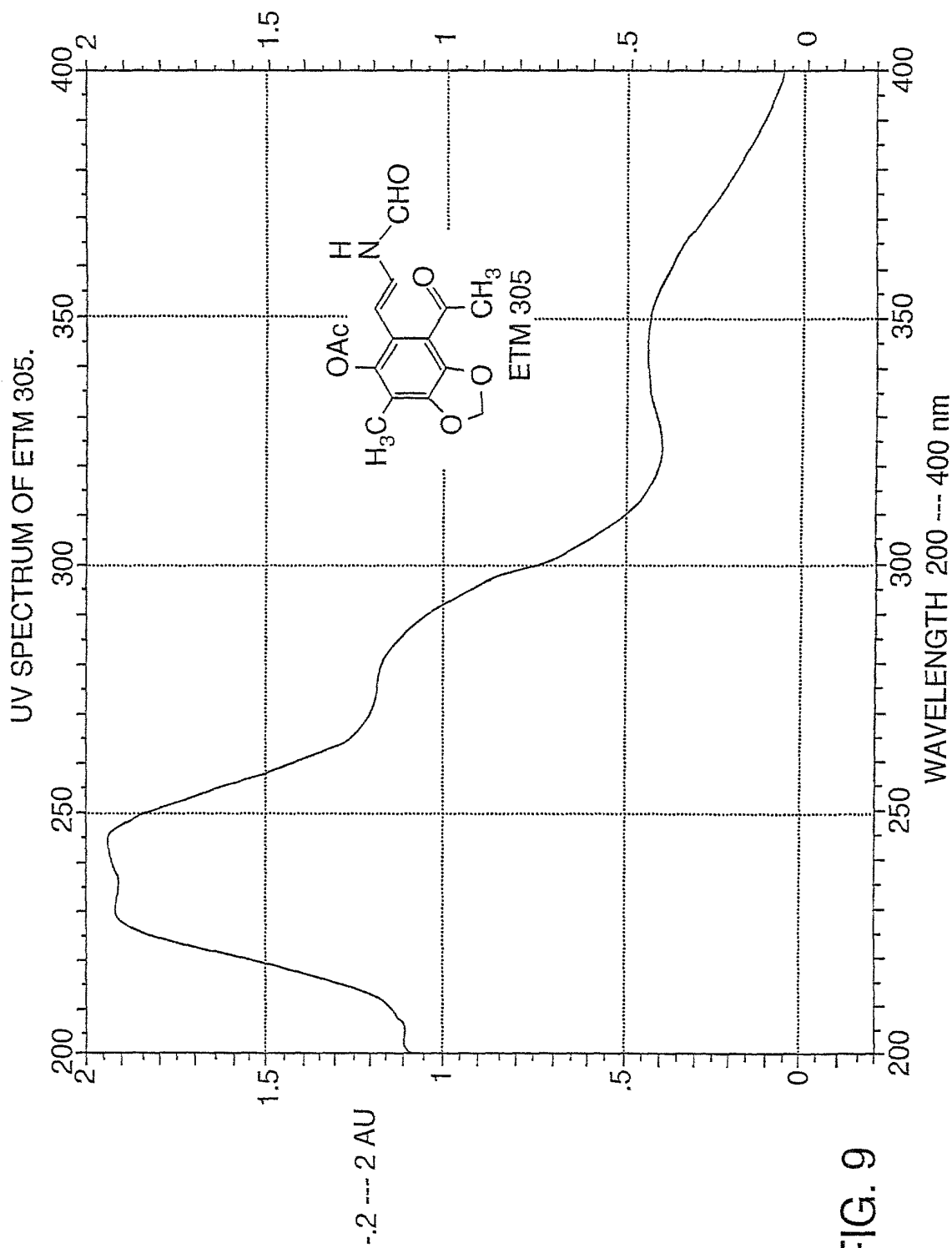


FIG. 9

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UV SPECTRUM OF ETM (PHARMAMAR).

INT OF WINDOW 39: UV APEX SPECTRUM OF PEAK 7.82 OF PICO-M2.D

UV. APEX SPECTRUM OF PEAK 7.82 OF PICO-M2.D

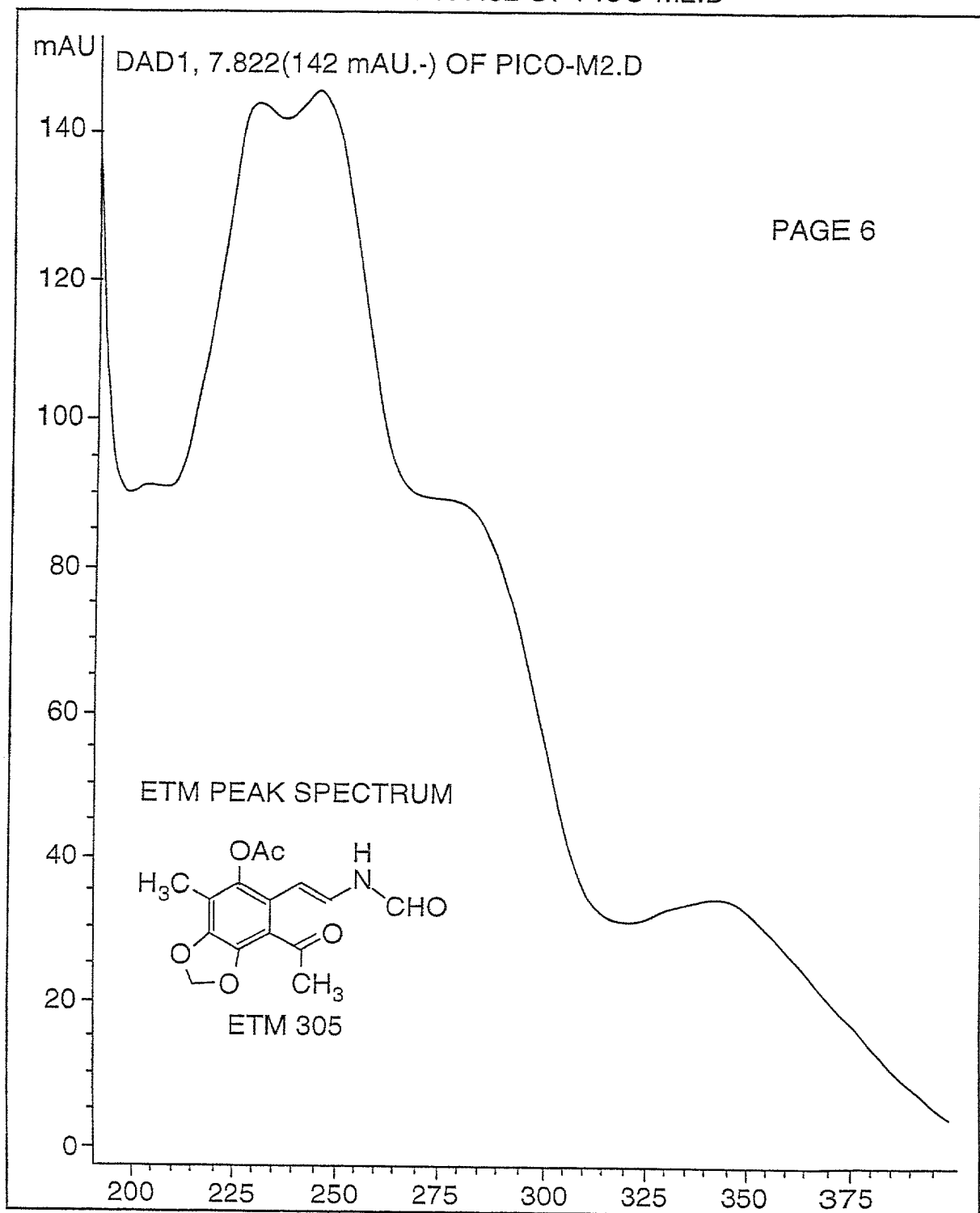


FIG. 10

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LRFAB MASS SPECTRUM OF ETM 775 IN M.B.

FILE: ETMSIOH5HP1 IDENT:2 ACQ: 16-DEC-1997 12:46:41 +1:00 CAL:CSI121697  
ZAB-SE FAB+ MAGNET BpV: 15.3V TIC:1626444160 FLAGS:HALL  
FILE TEXT: MORALES ETM-SIOH-5-HP1 IN M.B.

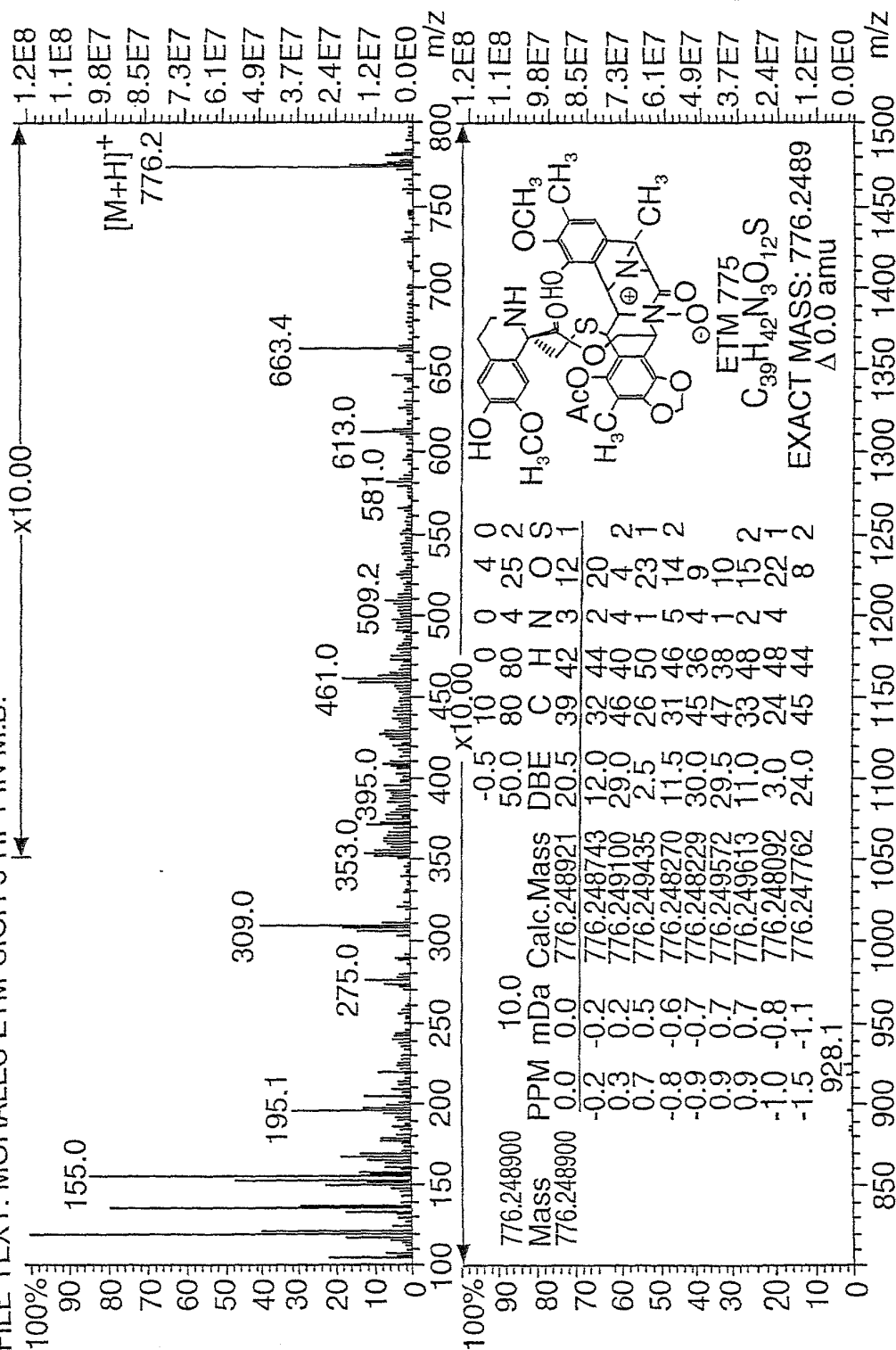
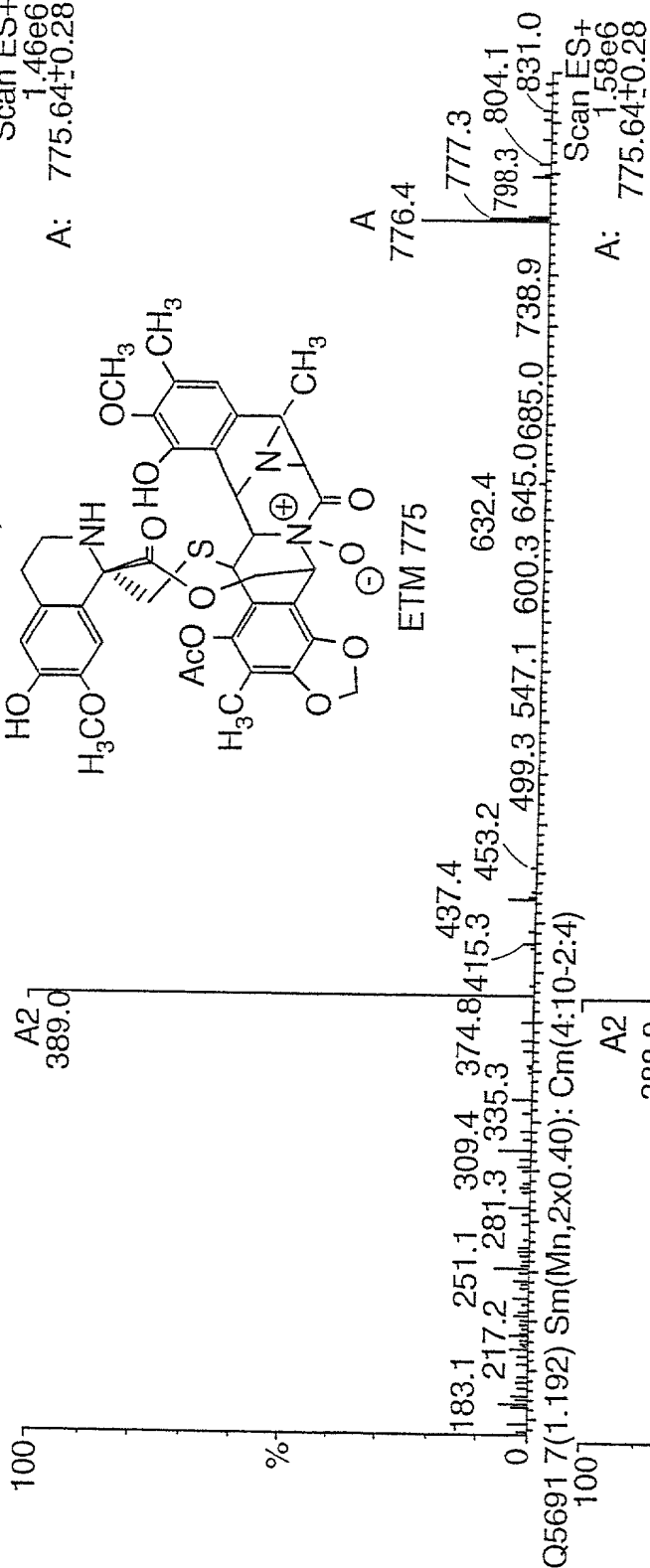


FIG. 11

## ESI MASS SPECTRUM OF ETM 775 (POSITIVE MODE).

etm-sioh5-hp1, 12/22/97

Q5691 7(1.192) Cn(Cen,6,80.00. Ht): Sm(Mn, 2x0.40): Cm(4:10-2:4)

Scan ES+  
1.46e6  
A: 775.64±0.28Scan ES+  
1.58e6  
A: 775.64±0.28

Q5691 7(1.192) Sm(Mn,2x0.40): Cm(4:10-2:4)

A2  
388.9  
[M/2+H]<sup>+</sup>[M+H]<sup>+</sup>  
A  
776.4  
777.4  
798.2  
m/z

FIG. 12

etm-775, 2/16/98

EYM775 7(1,194) Sm(Mn, 2x0.40): Cm (5:10-1:3)

Scan ES-  
1.07e5

ESI MASS SPECTRUM OF ETM 775 (NEGATIVE MODE).

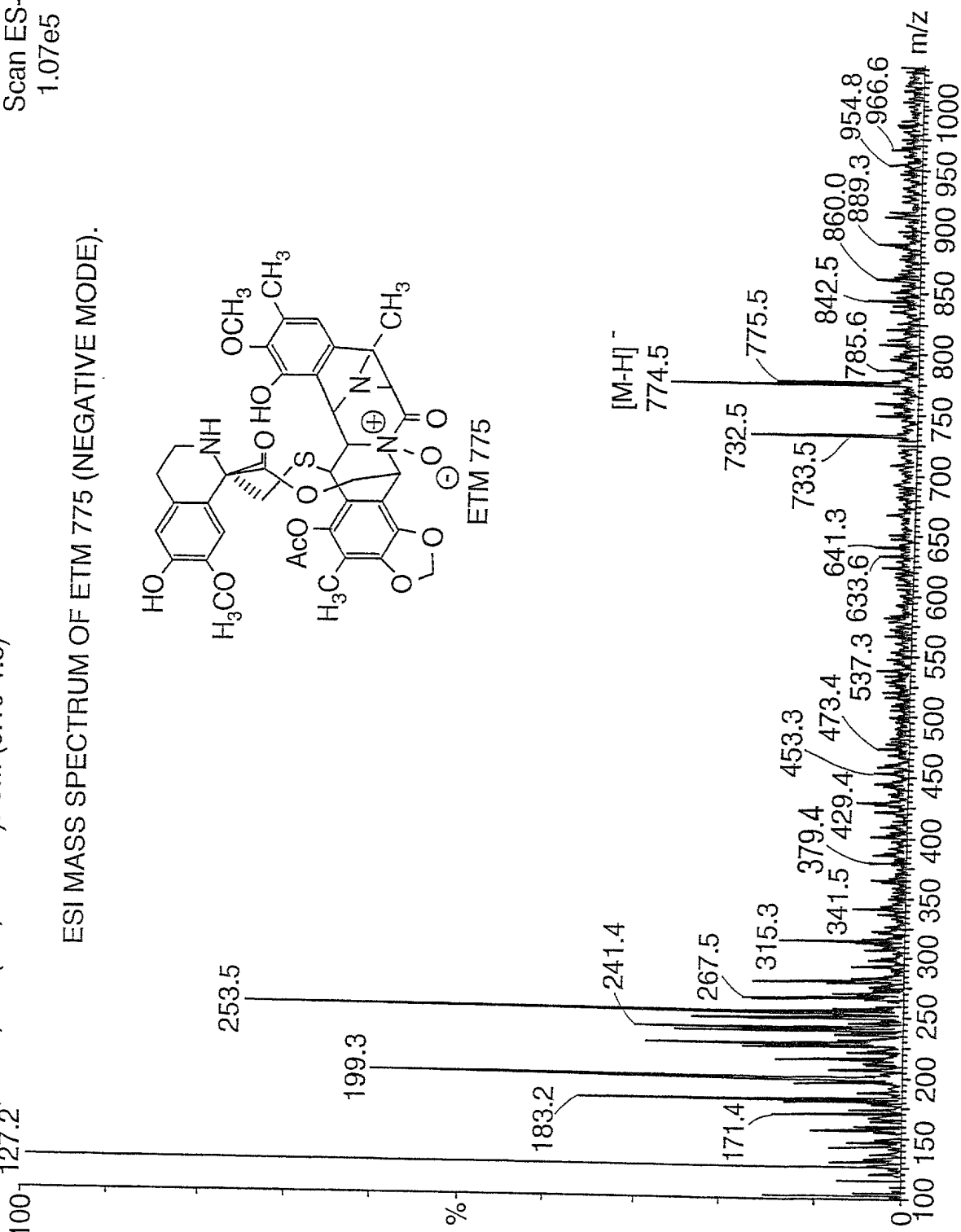
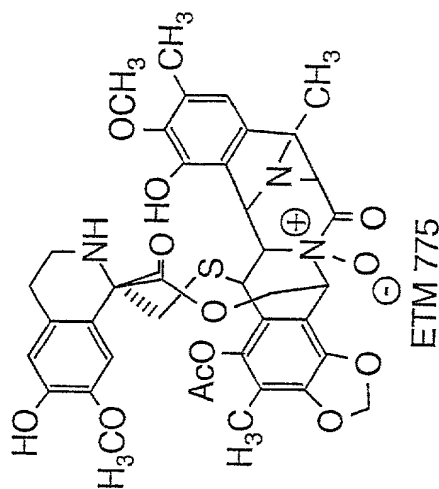


FIG. 13

FAB/MS/MS SPECTRUM OF ETM 775 ( $m/z$  138-302).

FILE: MS9082 IDENT:1 SMO(1,5) PKD (5,3,5,0.00%,0.0,0.00%,F,F) SPEC (HEIGHTS, CENTROID) ACQ:3-DEC-97&gt;

70-4SE FAB+ E2B2 BpM:777 BpV:146.3V TIC:371164064 FN:776.25 FLAGS:NORM

FILE TEXT: MORALES SFIM2275 MSMS ON 776.25 CELL 0.5 HE 90% MORALES SFIM2275 MSMS ON 776.25 CELL&gt;

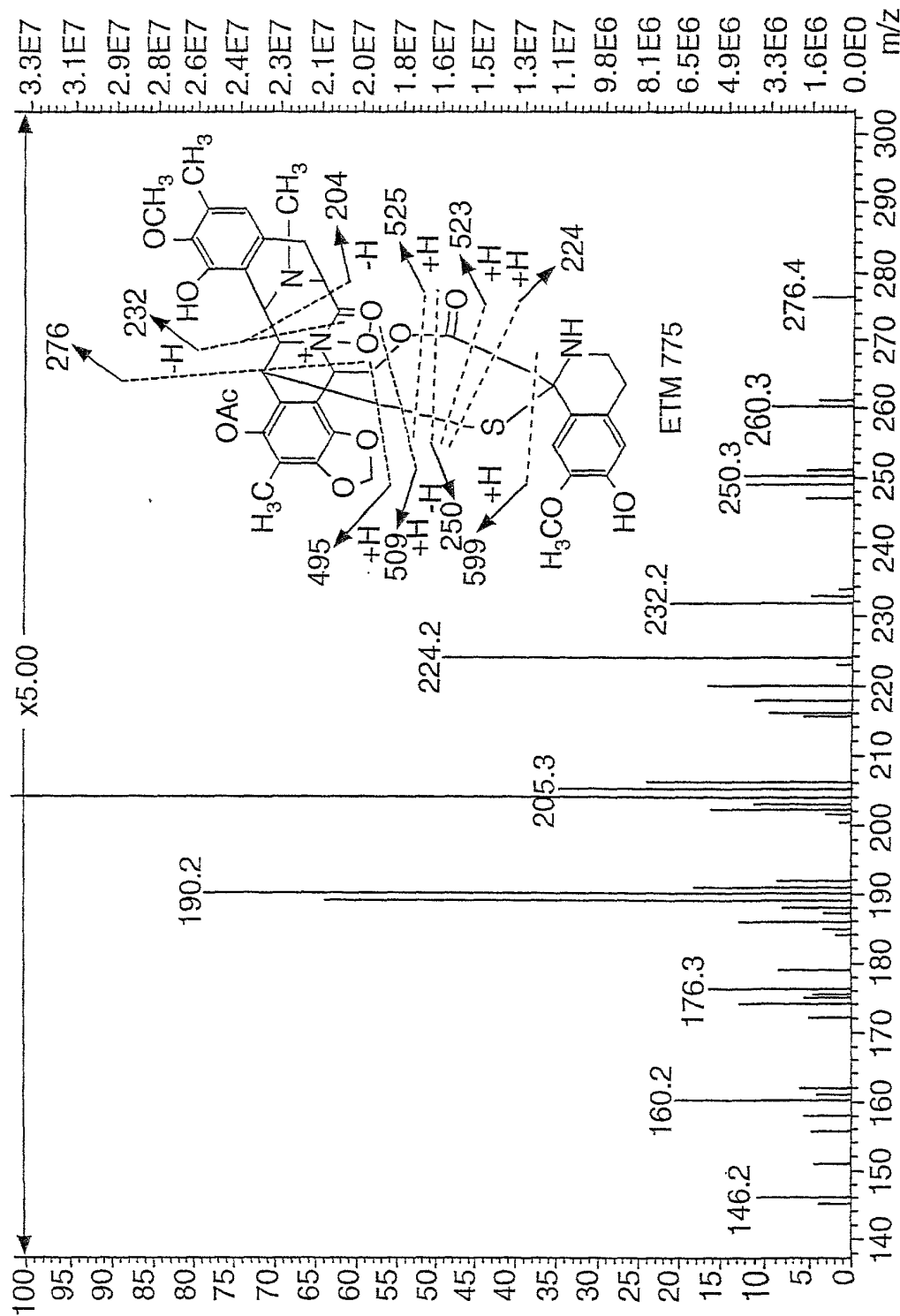
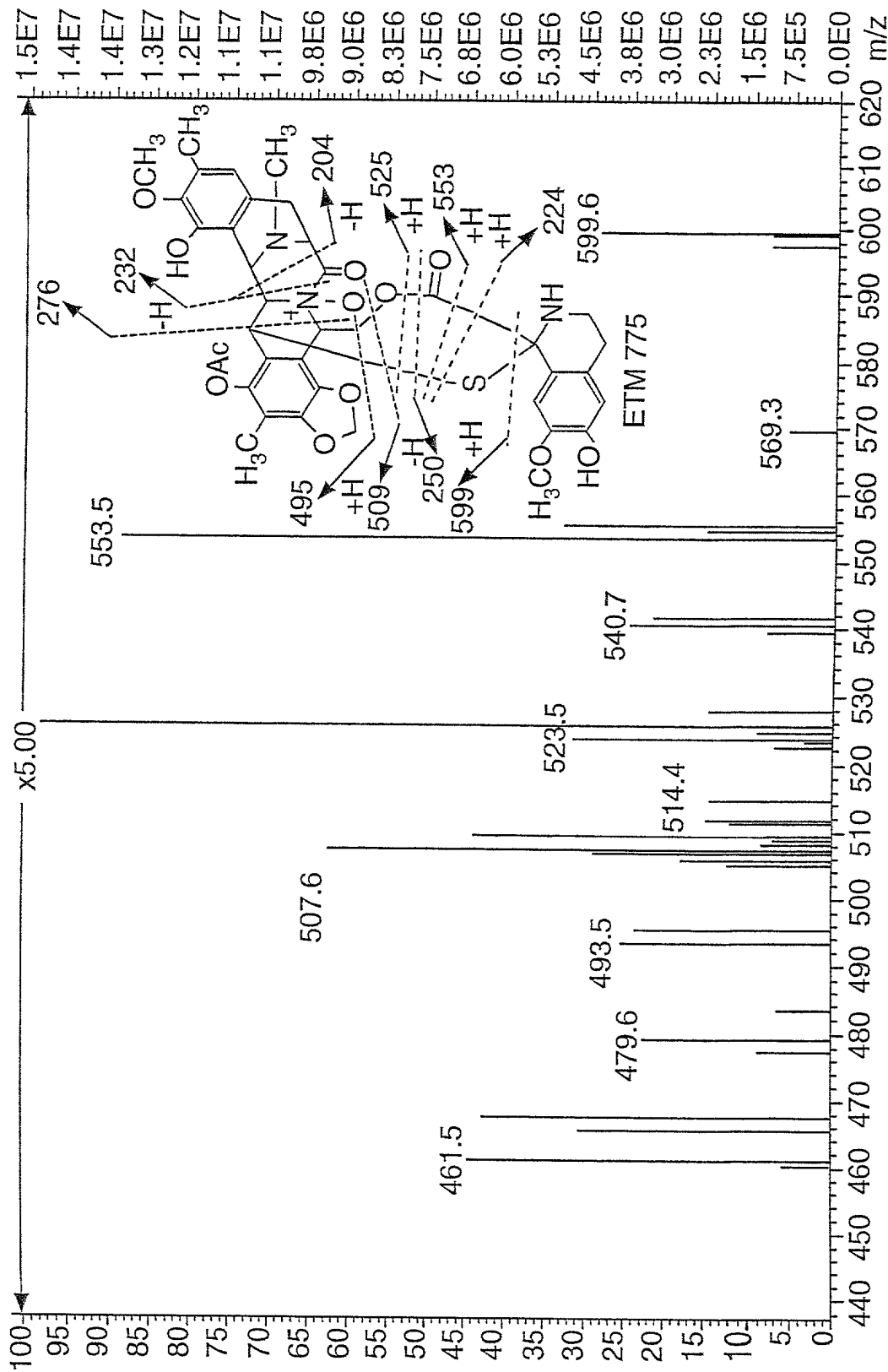


FIG. 14

FIG. 15

FILE: MS9082 IDENT:1 SMO(1,5) PKD (5,3,5,0.00%,0.0,0.00%,F,F) SPEC (HEIGHTS, CENTROID) ACQ:3-DEC-97>  
70-4SE FAB+ E2B2 BpM:777 BpV:146.3V TIC:371164064 FN:776.25 FLAGS:NORM  
FILE TEXT: MORALES SFIM2275 MSMS ON 776.25 CELL 0.5 HE 90% MORALES SFIM2275 MSMS ON 776.25 CE





<sup>1</sup>H NMR SPECTRUM (750 MHz) OF ETM 775 IN CD<sub>3</sub>OD.

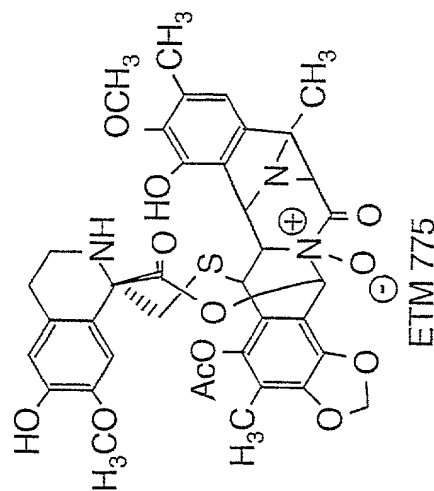
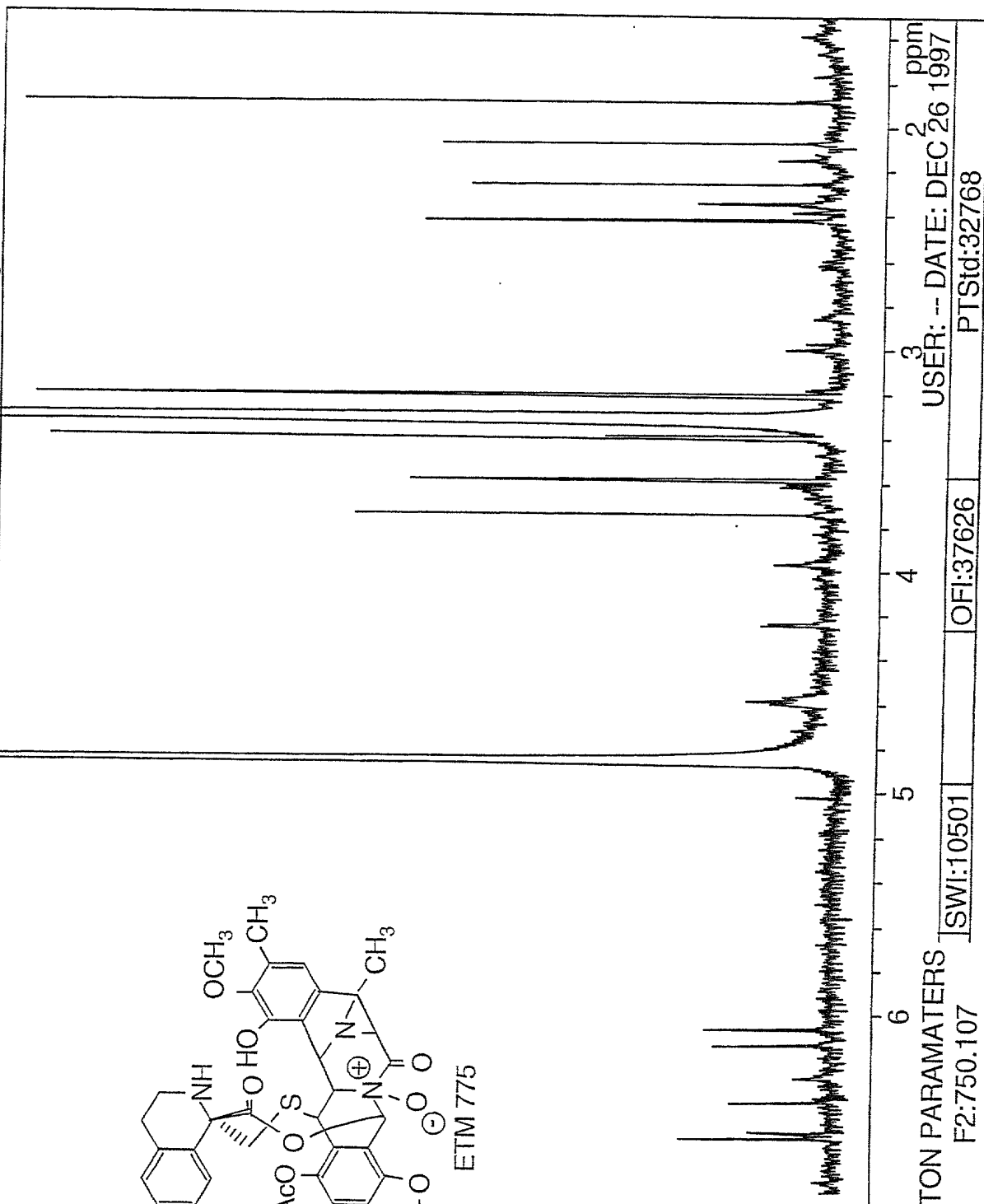


FIG. 16



DARD PROTON PARAMETERS

0.107

F2:750.107

SWI:10501

OFI:37626

PTStd:32768

3 2 ppm

USER: -- DATE: DEC 26 1997

UV SPECTRUM OF ETM 775.

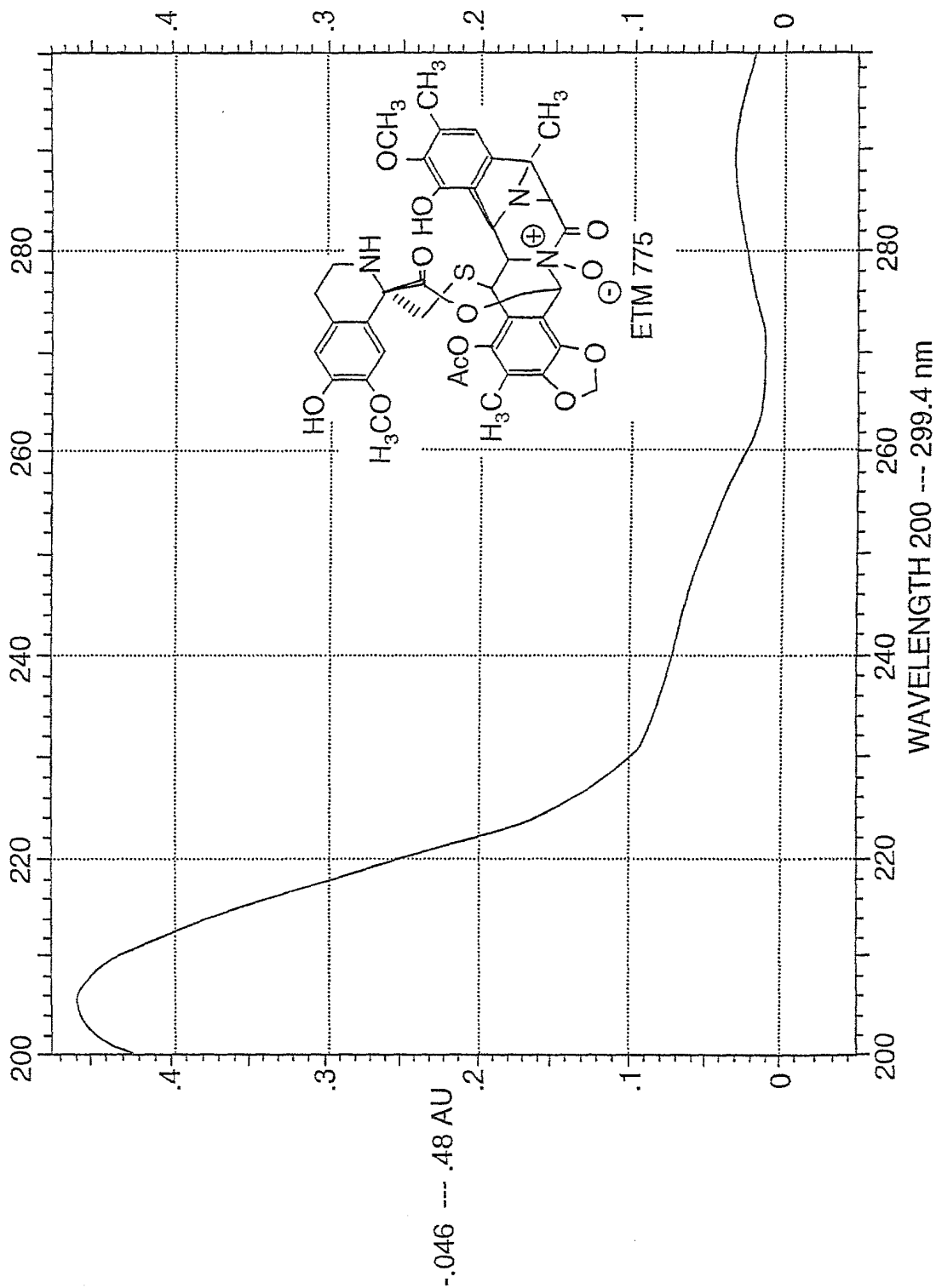


FIG. 17

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HPLC CHROMATOGRAM OF M1 METABOLITE (ETM 305).

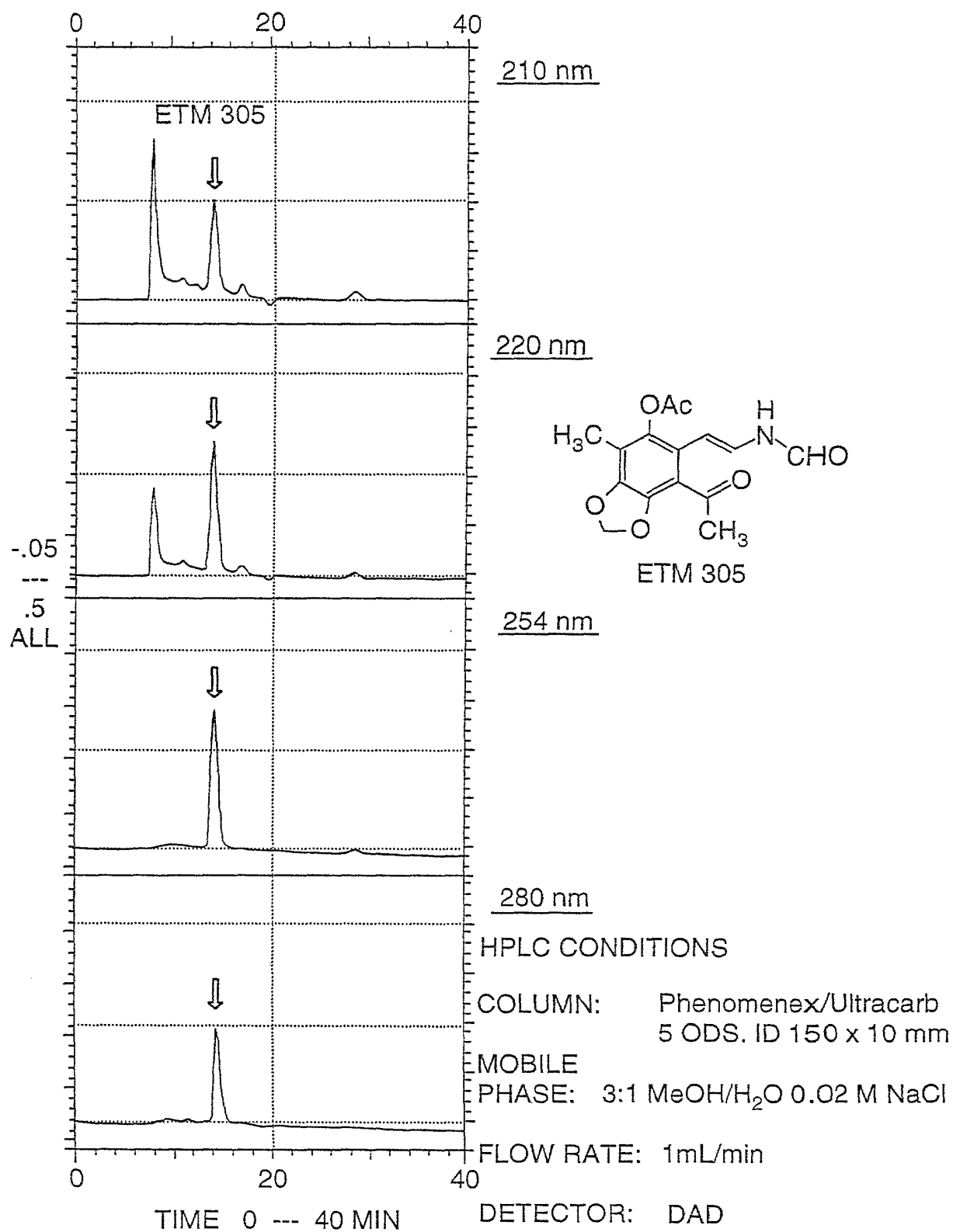
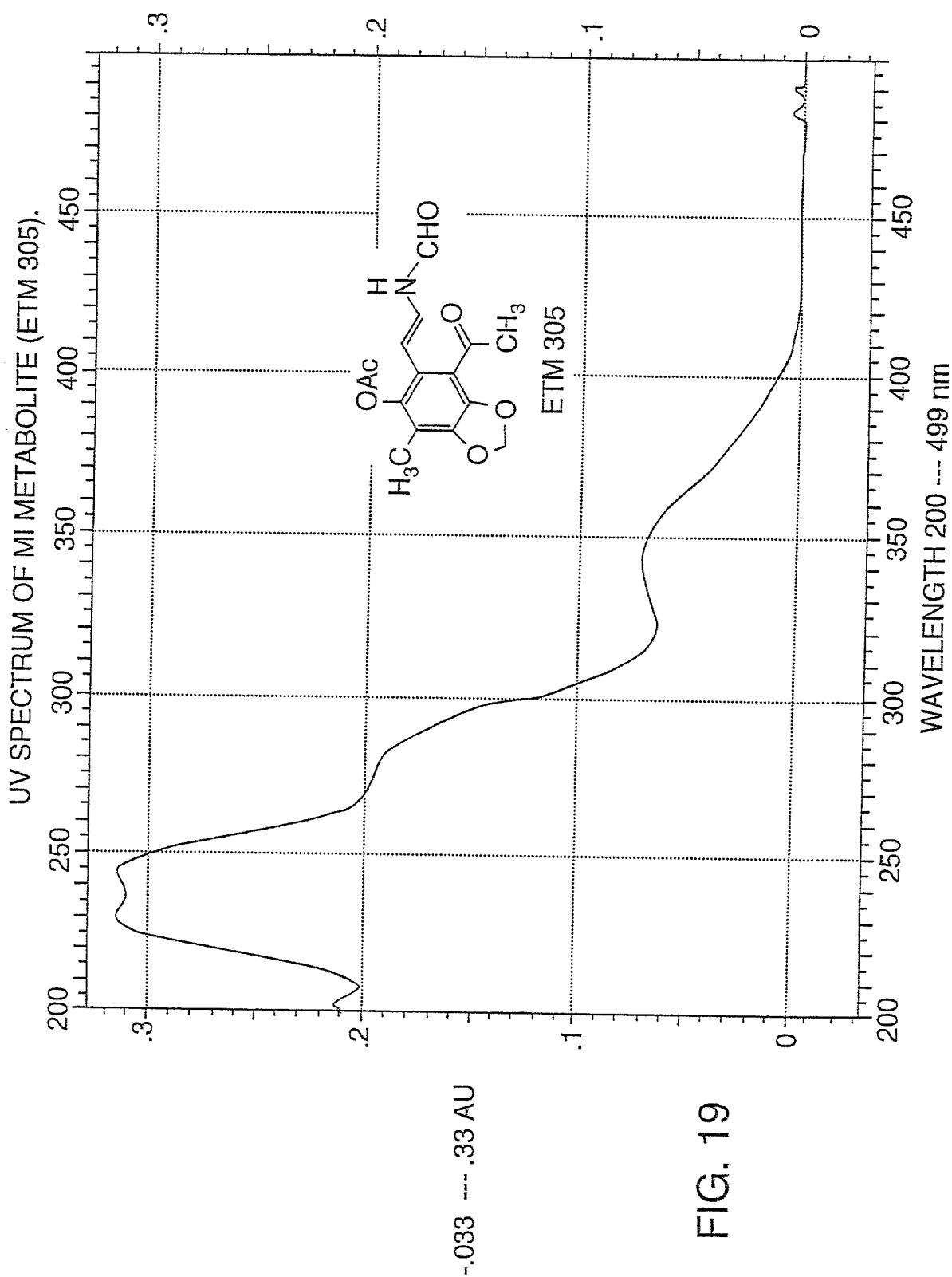


FIG. 18

[illegible]

ESI MASS SPECTRUM OF M1 METABOLITE (ETM 305).

M1-ODS-2. 4/1/98

M1ODS2 10 (1.692) Sm(Mn,2x0.40): Cm(6:14-1:4)

Scan ES+  
3.71e5

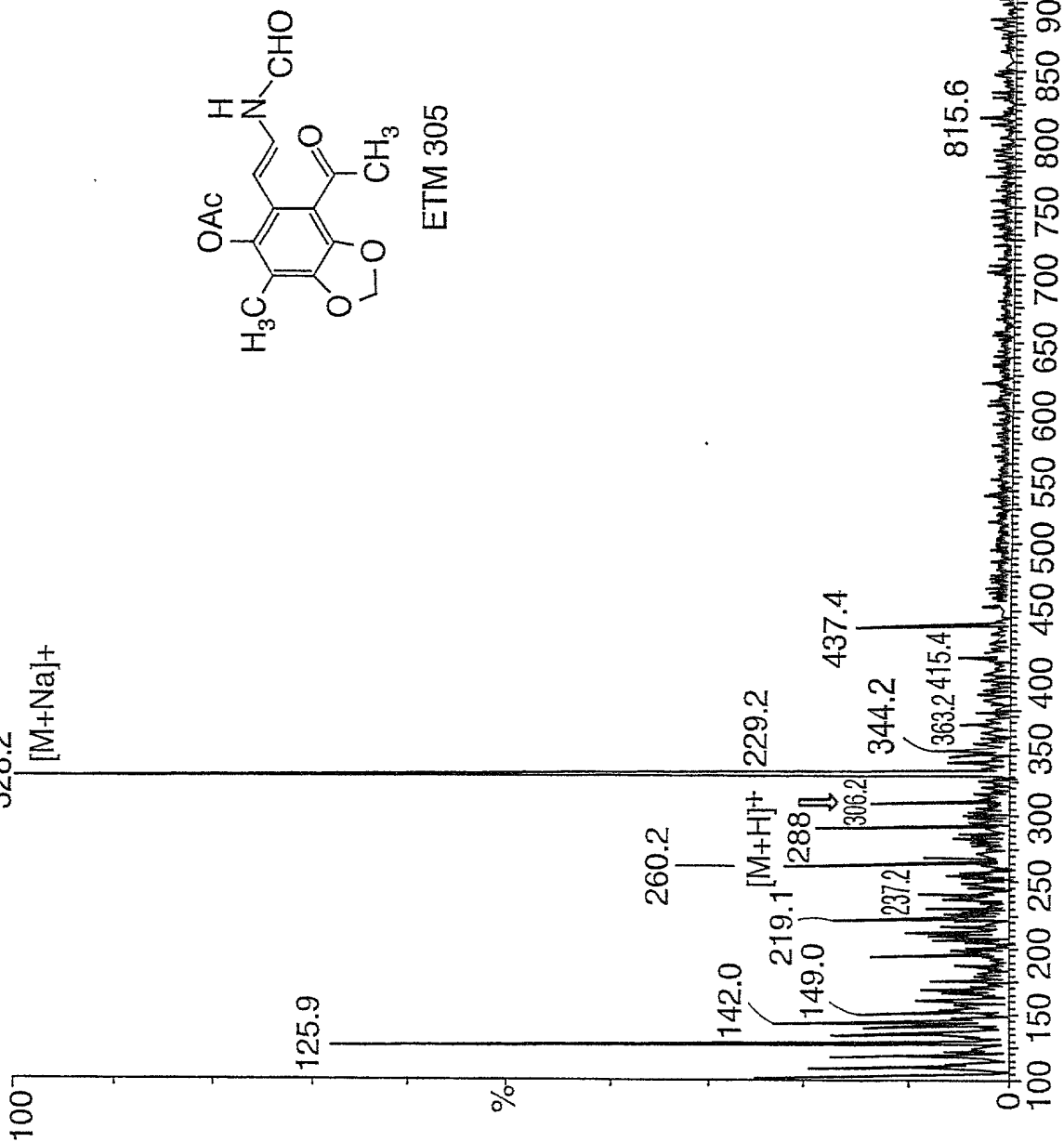


FIG. 20

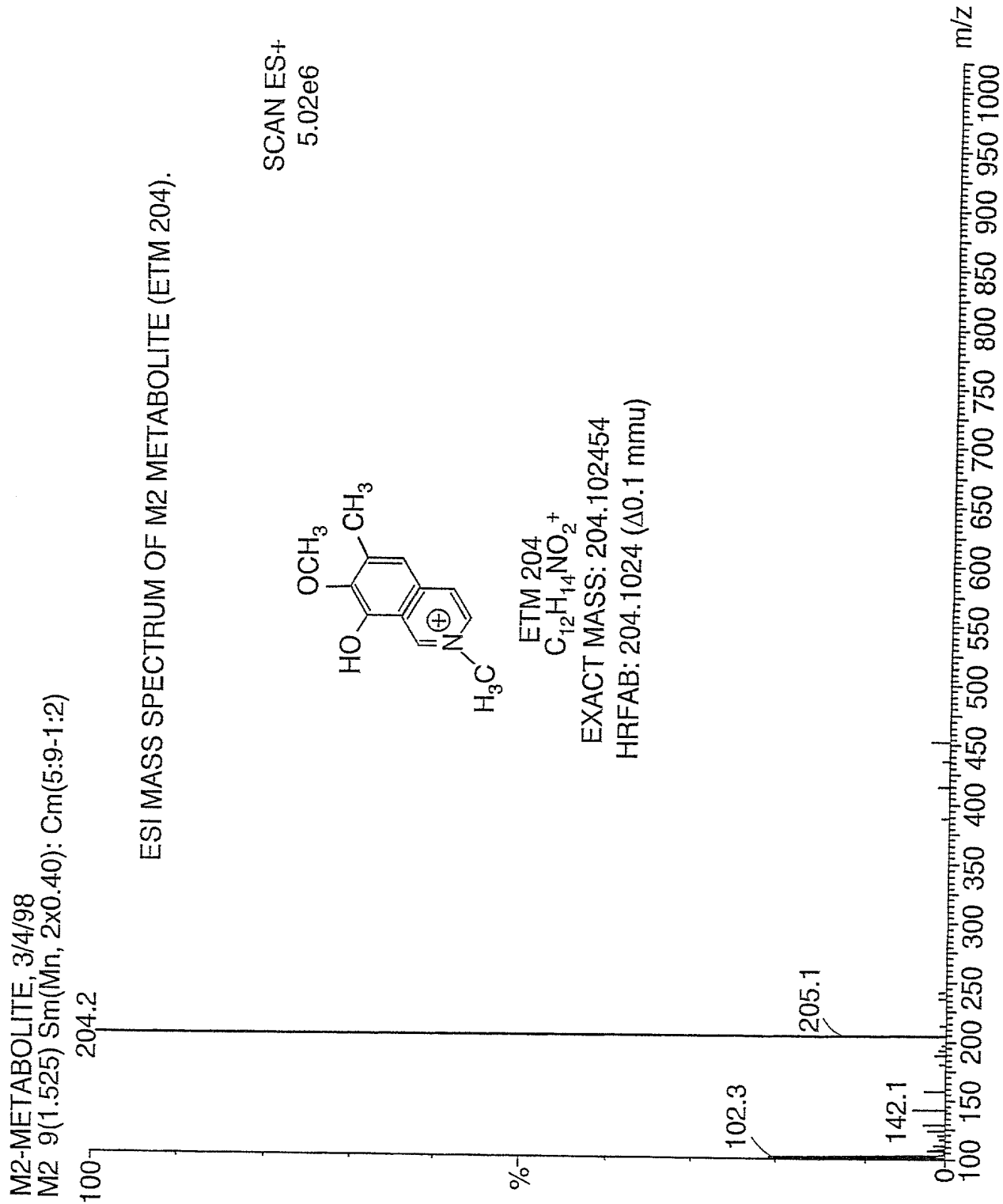


FIG. 21

FIG.  
22AFIG.  
22B

FIG. 22

MORALES, KLR, MZ IN CD3OD

EXPL S2PUL

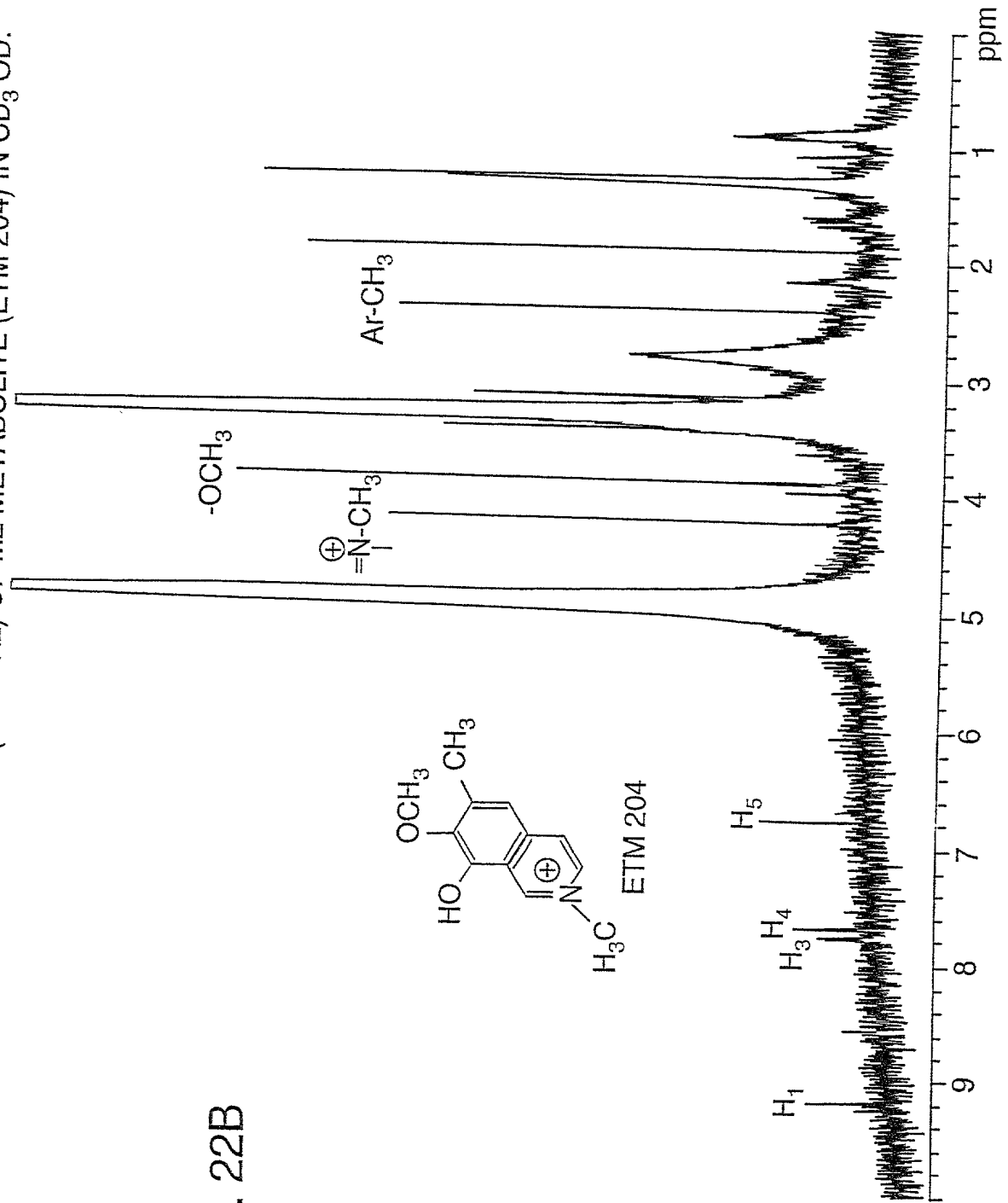
SAMPLE		DEC. & VT	
DATE	MAR 17 98	DFRQ	499.701
SOLVENT	METHANOL	DN	H1
FILE	EXP	DPWR	20
ACQUISITION		DOF	0
STFRQ	499.701	DM	NNN
TN	111	DMM	C
AT	4.003	DMF	200
NP	48000	DSEQ	
SW	5996.1	DRES	1.0
FB	3400	HOMO	N
BS	16	DEC2	
TPWR	63	DFRQ2	0
PW	4.5	DN2	
DL	0	DPWR2	1
TOF	0	DOF2	0
NT	3000	DM2	N
CT	1044	DMM2	C
ALOCK	N	DMF2	200
GAIN	NOT USED	DSEQ2	
FLAGS		DRES2	1.0
11	N	HOMO2	N
LN	N	LB	PROCESSING
DP	Y		0.30
HS	NN	WTFILE	
DISPLAY		PROC	FT
SP	-0.1	FN	NOT USED
WP	4997.0	MATH	F
V\$	31752		
SC	0	WERR	
WC	250	WEXP	
HZMM	19.99	WBS	
LS	33.57	WNT	
RFL	2154.5		
RFP	1649.0		
TH	7		
INS	1.000		
NM	PH		

FIG. 22A

09971853-100301  
10E00T-255T/660

<sup>1</sup>H NMR SPECTRUM (500 MHz) OF M2 METABOLITE (ETM 204) IN CD<sub>3</sub> OD.

FIG. 22B





ESI/MS/MS SPECTRUM OF M2 METABOLITE (ETM 204).

M1, DAUGHT OF 204.2

M2D 1(0.109) Sm(Mn, 2x1.00): Cm(1:12)

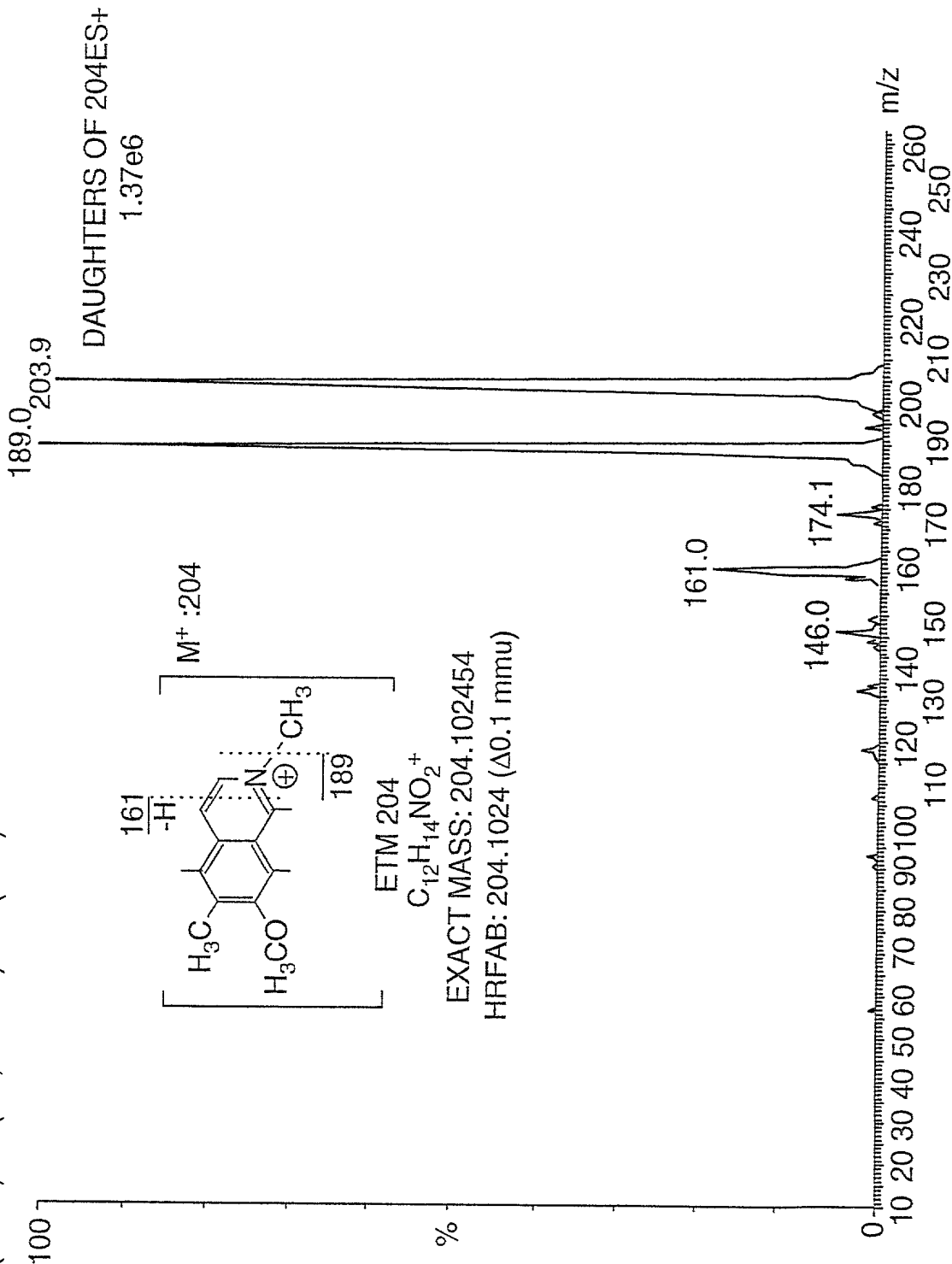


FIG. 23